

**Industrial Research at NSLS-II**  
**A Workshop to Showcase NSLS-II Capabilities and Create Industry Partnerships**  
**April 8-9, 2014**  
**Brookhaven National Laboratory**

**Industry/University Cooperative Research at a  
DOE Synchrotron Light Source**

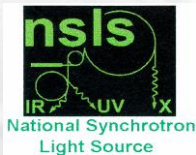
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**Acknowledgements** Work supported under Dow Corning. Topography experiments were carried out at the Stony Brook Synchrotron Topography Facility, Beamline X-19C, at the NSLS, BNL (DOE Office of Basic Energy Sciences Contract no. DE-AC02-76CH00016) and Beamline 1-BM at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL).

**Stony Brook Synchrotron  
Topography Facility  
Beamline X-19C**



*We help you  
invent the future.™*



**Department of Materials  
Science & Engineering**

# Outline

1c TSDs

## ● Our Working Model for Industry/University Collaborative Research at a DOE Synchrotron Light Source:

- Long Term Contracts.
- Short Term Projects
- Students Graduated.
- Papers Published

## ● Case Studies:

- Defect Density Reduction in SiC; Relaxation during Homo-Epitaxy :  
Funded by Cree; II-VI Inc.; Dow Corning
- Defect Analysis in Sapphire: Funded by St. Gobain; ARC Energy
- Defect Analysis in PVT Grown AlN Crystals: funded by Hexatech Inc.

## ● NSLS-II HXT Beamline Development Proposal Approved 2013

- Possible collaboration with MID beamline (NxtGen) along with MDM.

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# Industry/University Cooperative Research at a DOE Synchrotron Light Source

## What is our Working Model?

● Our expertise is in characterizing crystallographic defects in single crystals and determining their origins – long term goal of engineering the defect densities to promote improved performance of devices fabricated on the crystals

● >25 years of experience

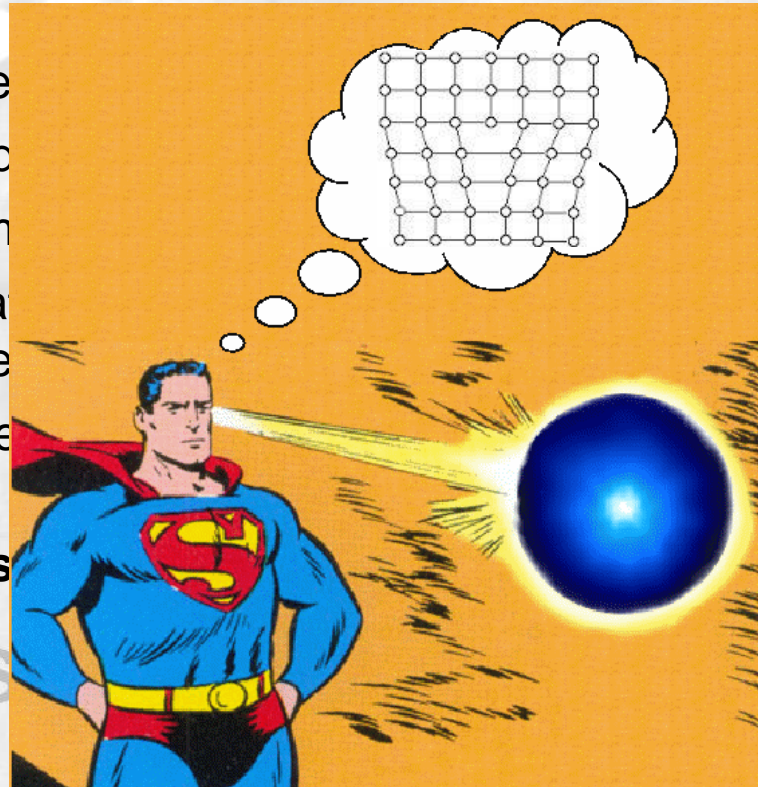
● Collaborative Model

● Problems of common interest

● Synchrotron radiation used to achieve industry goals while advancing basic science

● Non-proprietary research results shared with science community

● We are the “eyes and ears” of the research community



● Sponsored research

● Partner

● Information critical to joint papers

● Significant contributions to the field

●

- Our long established track record encourages trust on the part of the industrial collaborator
- Projects can be short term – company contributes to maintenance and support of the beamline (covers consumables, technical support, etc)
- Or longer term through a contract awarded to our Research Foundation which pays for manpower (graduate students, postdocs) and also contributes to the maintenance and support of the beamline)
- Can be directly funded by industrial partner or by subcontract from a federal grant
- Since 1987, we have worked with numerous companies. Most often they contact us.
- Graduated some 16 Ph.D students on industrially sponsored projects
- Generated some 140 peer reviewed publications (out of a total of some 360) based on industrially sponsored research



## Ph.D Students and Postdocs

G.-D. Yao (Ph.D 1992, Daqo New Energy); J. Wu (Ph.D 1992, Intel); S. Wang (Ph.D 1995, Fairfield Crystals); T. Fanning (Ph.D 1996, Avago Technologies); H. Chung (Ph.D 1998, Applied Materials); D. Bliss, (Ph.D, 2000 Airforce Research Lab., Hanscom); W.M. Vetter (Ph.D, 1999); B. Raghothamachar (Ph.D 2001); Huaibin Chen (Ph.D 2005, Research Scientist, NYU School of Medicine); J. Bai (Ph.D 2006, Intel, OR); G. Wang (Ph.D 2007 Saint Gobain); Y. Chen (Ph.D 2008, Western Digital); Hui Chen (Ph.D 2008, Headway Technologies); Yu Zhang (Ph.D 2011, Global Foundries); V. Sarkhar (Ph.D 2012, Kyocera); S. Byrappa ((Ph.D 2014, Global Foundries);

*H. Wang, (PhD candidate since 2009); F. Wu (PhD candidate since 2009); J. Guo (PhD candidate since 2014); O. Goue (PhD candidate since 2013); Y. Yang (PhD candidate since 2013);*

Dr. X. Huang, Postdoc with me, 1996 – 2006, APS, ANL; W.M. Vetter, Postdoc. with me 1999-2006, Sensitron; Dr. G. Dhanaraj, Postdoc with me, 2000 – 2007, Aymont Technologies; B. Raghothamachar, Postdoc with me 2001-2010, Research Professor 2010-

# Case Studies

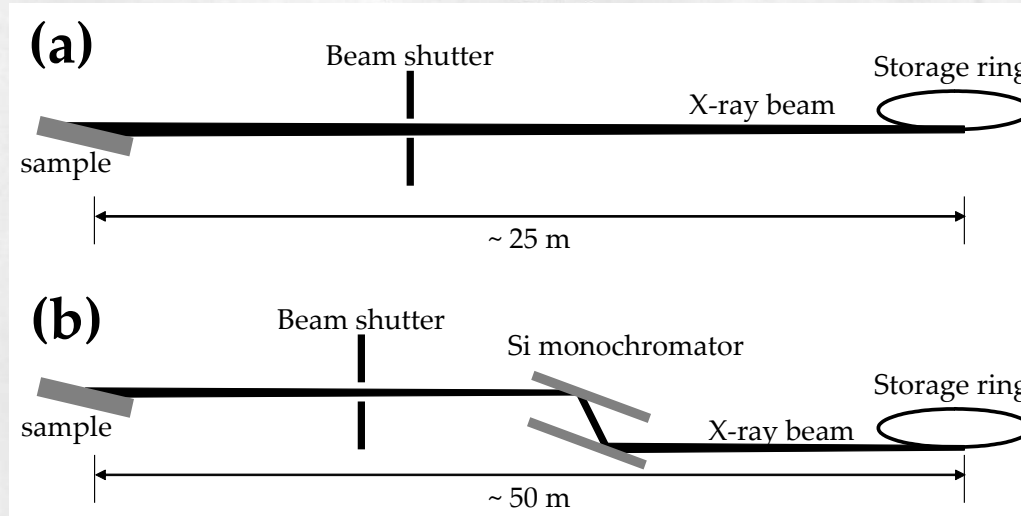
- Defect Density Reduction in SiC; Relaxation during Homo-Epitaxy
- Defect Analysis in Sapphire
- Defect Analysis in PVT Grown AlN Crystals

1c TSDs

TEDs

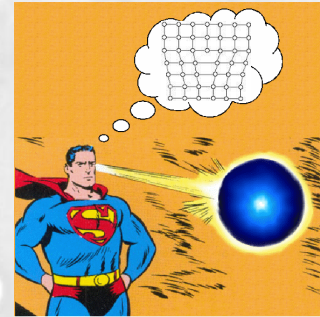
TEDs

# Synchrotron x-ray topography



NSLS

APS



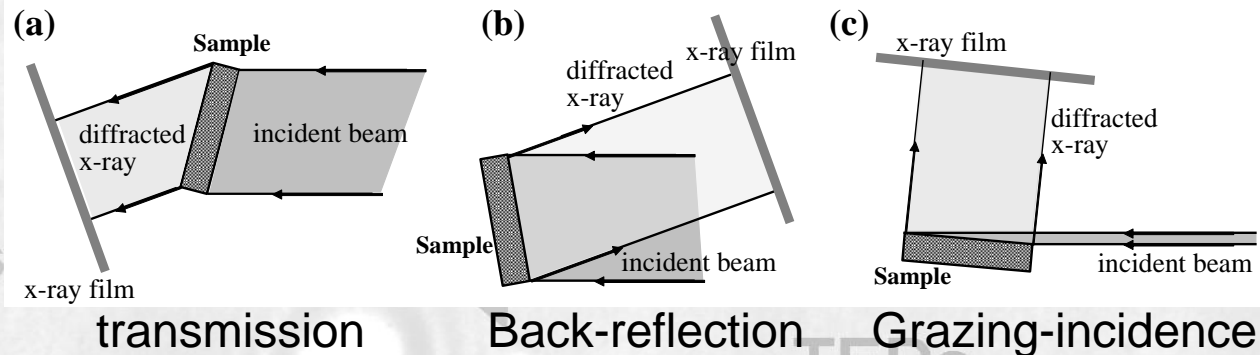
topographic resolution ( $R_x$ ):  $R_x = d S_x / D$ .

the source size in the incidence plane ( $S_x$ ), source-specimen distance ( $D$ ) and the specimen-film distance ( $d$ )

$d = 10$  cm:

~ 0.4  $\mu\text{m}$  for NSLS-X19C

~ 0.06  $\mu\text{m}$  for APS -1-BM



transmission

Back-reflection

Grazing-incidence



# Why SiC?

**1. Silicon carbide (SiC) is gradually replacing conventional semiconductor materials (e.g., Si/GaAs) due to its:**

- high breakdown field ( $2.5 \times 10^6$  V/cm, Si:  $\sim 3 \times 10^5$  V/cm)
- high thermal conductivity (4.9 W/cm K, compared with Si: 1.3 W/cm K)
- high saturated electron drift velocity ( $2 \times 10^7$  cm/sec, Si:  $1 \times 10^7$  cm/sec)
- large band gap (4H-SiC: 3.2 eV; 6H-SiC: 3.0 eV; Si: 1.12 eV)
- good mechanical and chemical stability
- high resistance in radiation environment

**The unique combination of these properties of SiC makes it an ideal material for high-temperature, high-power and high-frequency applications: high speed power switch, high temperature thyristors...**

	$E_g$ (eV)	$T_w$ (K)	$k$ (W/cm·K)	$\mu_e$ (cm <sup>2</sup> /V·s)	$\mu_h$ (cm <sup>2</sup> /V·s)	$E_b$ (10 <sup>5</sup> V/cm)	$V_s$ (10 <sup>7</sup> cm/s)
Si	1.12	410	1.5	1400	600	2	1
6H-SiC	3.0	1200	5	370	90	37	2

$E_g$ : band gap;  $T_w$ : working temperature;  $k$ : thermal conductivity;  $\mu_e$ : electron mobility;  
 $\mu_h$ : hole mobility;  $E_b$ : breakdown field;  $V_s$ : saturated carrier velocity.

**2. In addition, SiC is widely used as substrates for nitride-based light emitting diodes (LEDs) due to its smaller lattice mismatch (~3.4%) compared with sapphire (~16%).**



# Benefits of SiC:

## **Spacecraft:** *Solar System Exploration*

Spacecraft with high temp., radiation hard SiC electronics will enable challenging missions in both the inner and outer solar system.



## **Aircraft:** *High Temperature Sensor and Control Electronics*

SiC electronics and sensors that could function mounted in hot engine and aerosurface areas of an aircraft would enable substantial weight savings, increased jet engine performance, and increased reliability.



## **Automobiles:** *High Temperature Sensor and Control Electronics*

SiC high temp. electronic sensors and controls on an automobile engine will lead to better combustion monitoring and control, which would result in cleaner burning, more fuel efficient cars.

### *High Power Electronics for Electric Vehicles*

SiC will enable more practical electric vehicles and other transportation systems by means of vastly improved SiC-based power electronic devices.



## **Communications:** *High Power, High Temperature Microwave RF Electronic Devices*

SiC based microwave electronics can function at large densities and high temperatures offering significant improvements to wireless communications and radar.



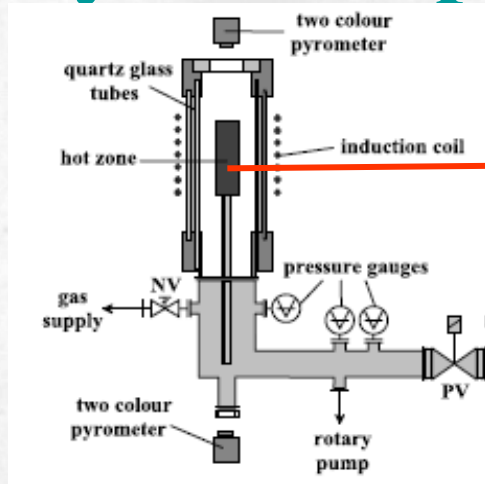
## **Power:** *Energy Savings in Public Power Distribution*

Superior SiC power electronics could increase the efficiency and reliability the public electric power distribution system.



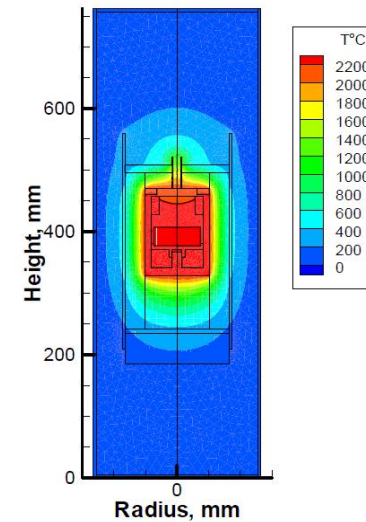
Source: <http://www.grc.nasa.gov/WWW/SiC/automobiles.html>

# Physical Vapor Transport (PVT) of SiC



Typical Induction Furnace  
Power=7-10 KW  
Pressure<300 T  
Temperature >2000 Celsius

1c TSDs  
Reaction Cell



Model of Temperature Distribution in Furnace

- 4H n+ SiC crystals were grown using physical vapor transport (PVT) on a 4H SiC, C-face seed.
- A standard graphite crucible was used to grow both 3" and ~4" diameter 4H n+ SiC crystals.
- The growth temperature was between 2000 and 2300° C at sub-atmospheric pressures.
- N<sub>2</sub> and Ar gas were added to the growth chamber to control resistivity.
- Growth rates for all investigations were between 0.1 and 1 mm/hr.



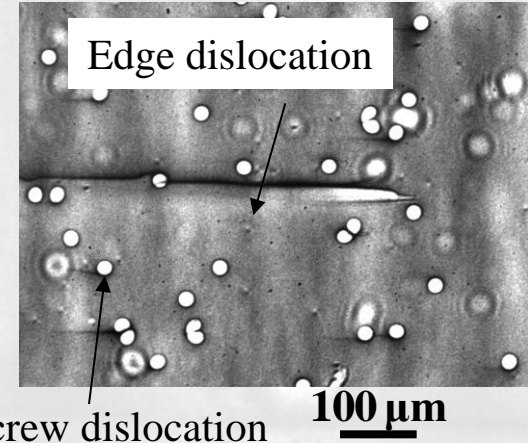
## Motivation – current issues

- In bulk/epitaxial material: high defect density compared to conventional semiconductor materials.

Si: dislocation free  $0/\text{cm}^2$ ;

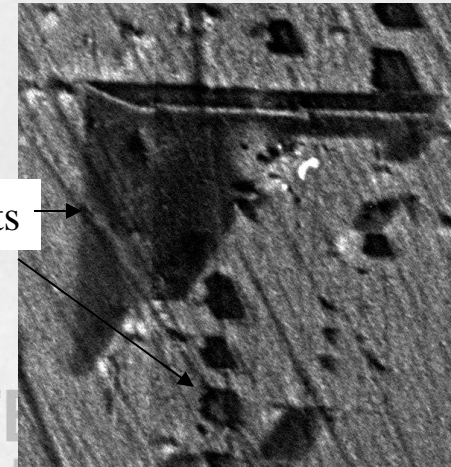
SiC: total dislocation density  $\sim 10^4/\text{cm}^2$

- threading screw dislocations (TSDs)  $< 10^3/\text{cm}^2$ ;
- threading edge dislocations (TEDs)  $\sim 10^3\text{-}10^4/\text{cm}^2$ ;
- basal plane dislocations (BPDs)  $\sim 10^2\text{-} \text{few} \times 10^3/\text{cm}^2$ ;
- other defects (SFs, grain boundaries);



- All defects protruding into device active-areas have detrimental effect on device performance (pn junctions, Schottky, etc.). (P. Neudeck, W. Huang, and M. Dudley, *IEEE Trans. Electron. Devices*, **46**, 478-484; (1999). H. Chen, B. Raghothamachar, W. Vetter, M. Dudley, Y. Wang, B.J. Skromme, *Mater. Res. Soc. Symp. Proc.*, **911**, 0911-B12-03, 169-174, Warrendale, PA, (2006)).

- In epilayer structures fabricated for pin devices, electron-hole recombination enhanced dislocation glide (REDG) activates the glide of partial dislocations under forward electrical bias. This results in the expansion of basal stacking faults, causing forward voltage drop.



Stacking faults



# Threading Screw Dislocations (TSDs) and Micropipes (MPs) (Cree 6H-SiC Crystals circa 1995)

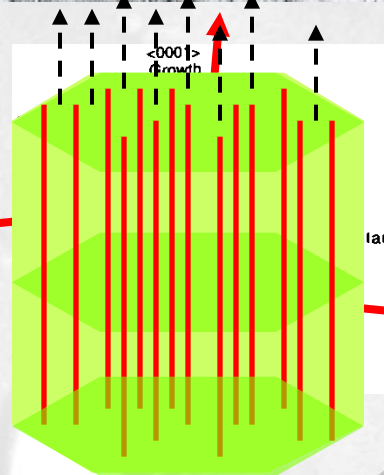
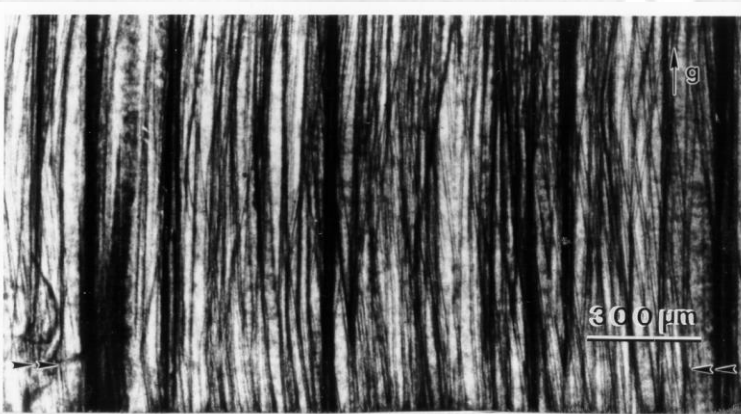
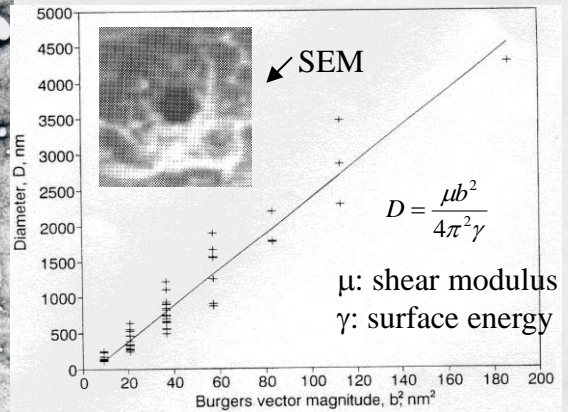
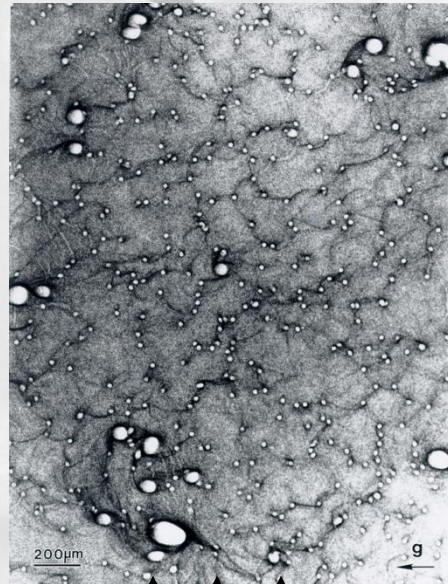
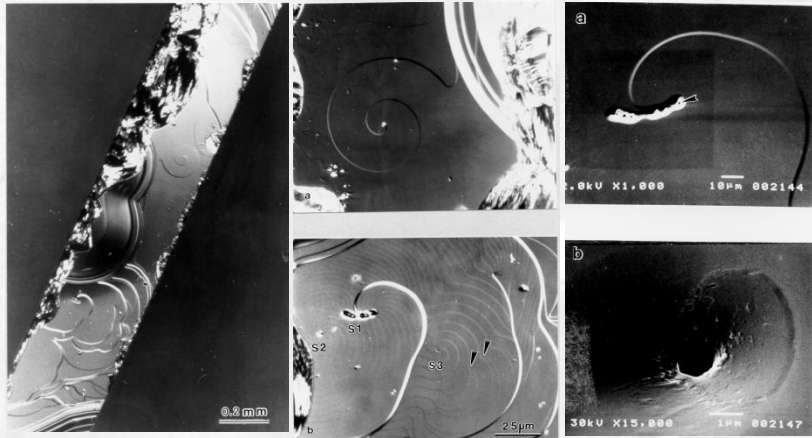


Image from longitudinal slice showing the seed/crystal interface region (indicated by arrows) in a 6H-SiC crystal grown by Physical Vapor Transport. One-to-one correlation can be found between [0001] screw dislocations in the seed and in the newly grown crystal. Dislocations of various Burgers vector can be observed ( $b=[0006]$  to  $b\sim 5[0001]$ ).

**TSD Density: 22,200 cm<sup>-2</sup>**

Similar image, lower defect density region.

**TSD Density 11,100. cm<sup>-2</sup>**

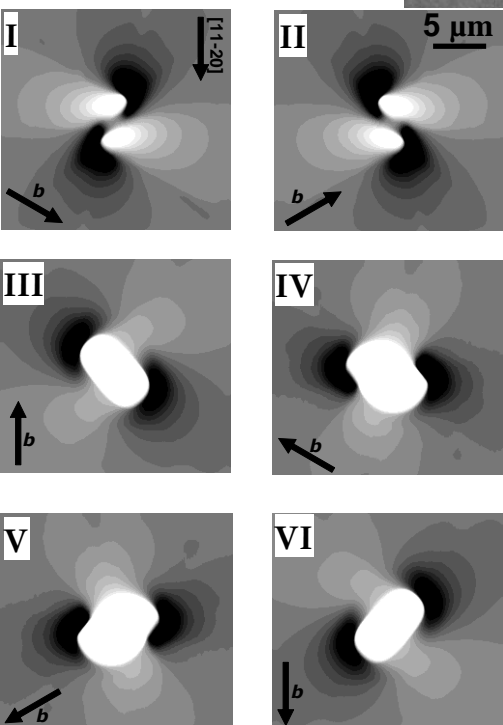


# Defect structures in SiC Substrates

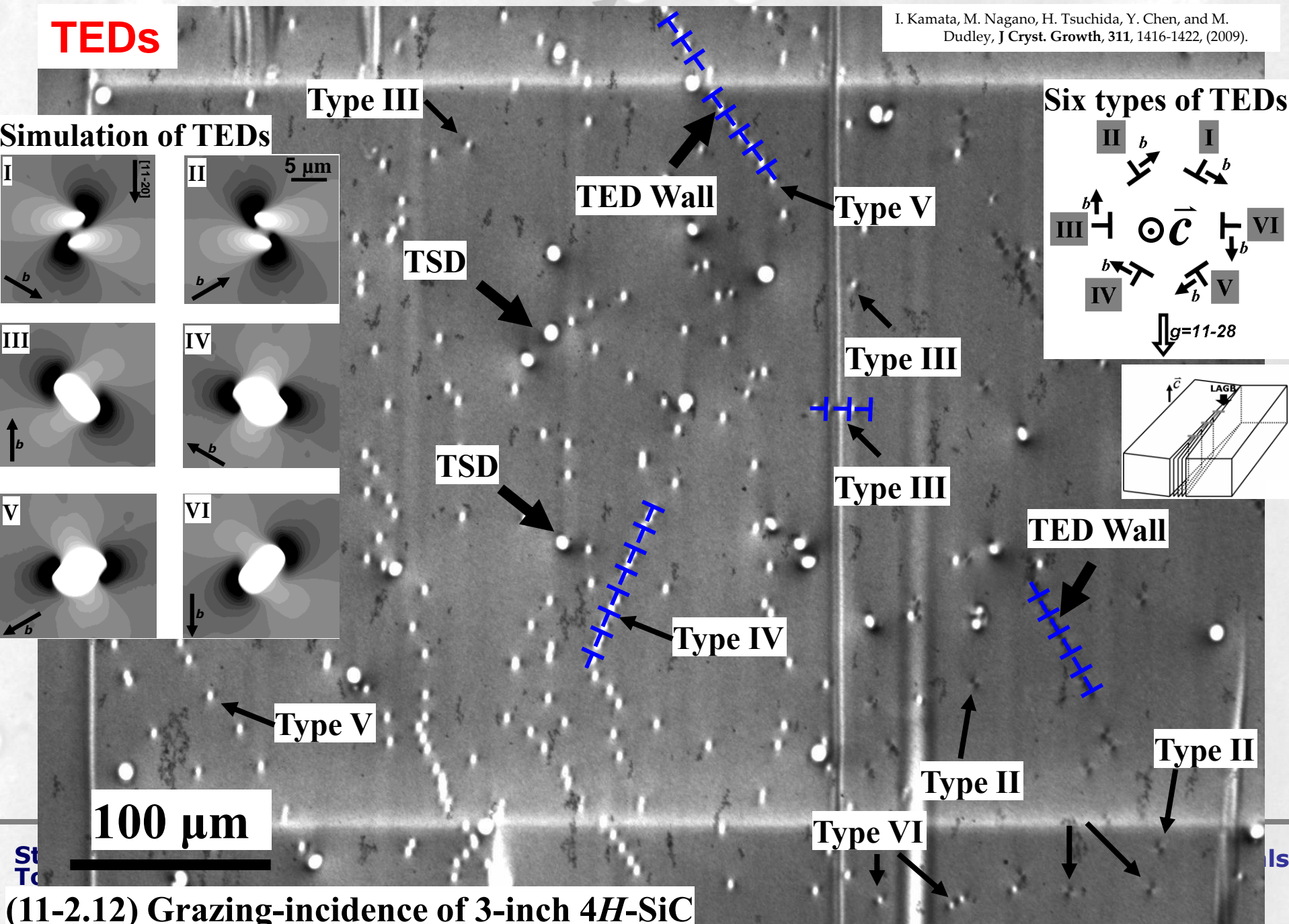
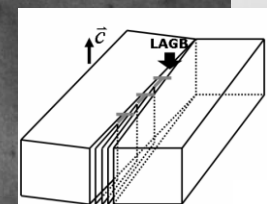
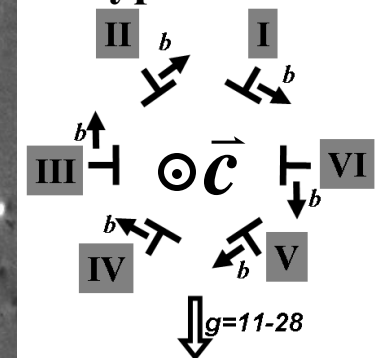
**TEDs**

I. Kamata, M. Nagano, H. Tsuchida, Y. Chen, and M. Dudley, *J Cryst. Growth*, 311, 1416-1422, (2009).

**Simulation of TEDs**



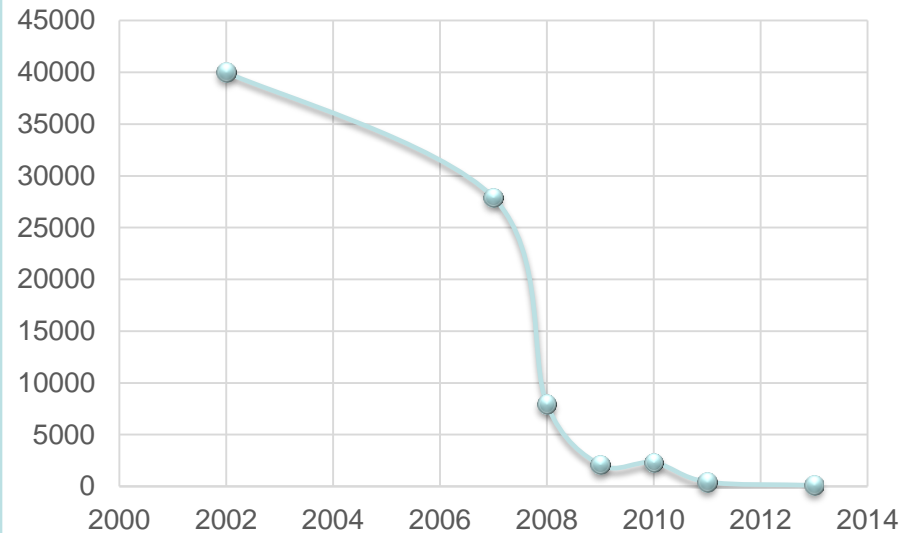
**Six types of TEDs**



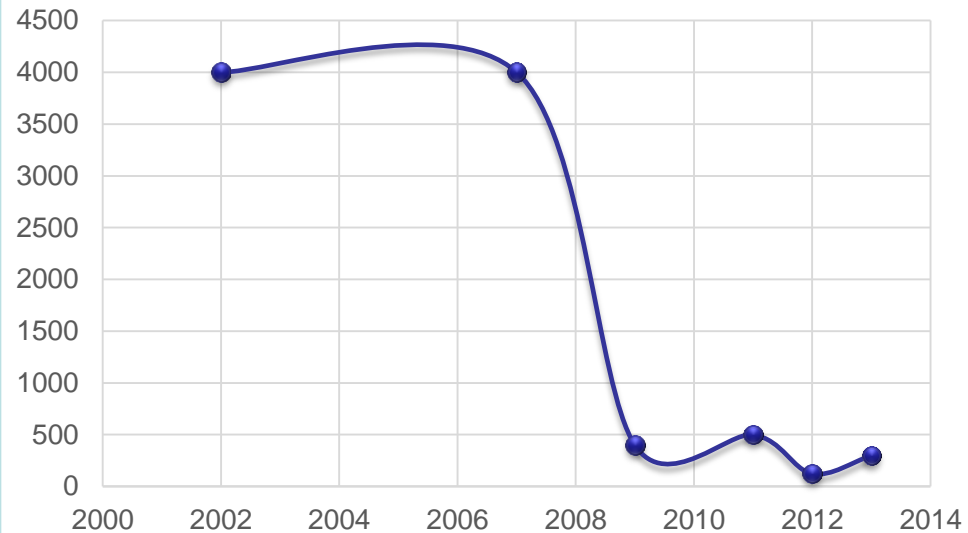
(11-2.12) Grazing-incidence of 3-inch 4H-SiC

# Substrate Dislocation Density Trends

## BPD Density(cm-2)



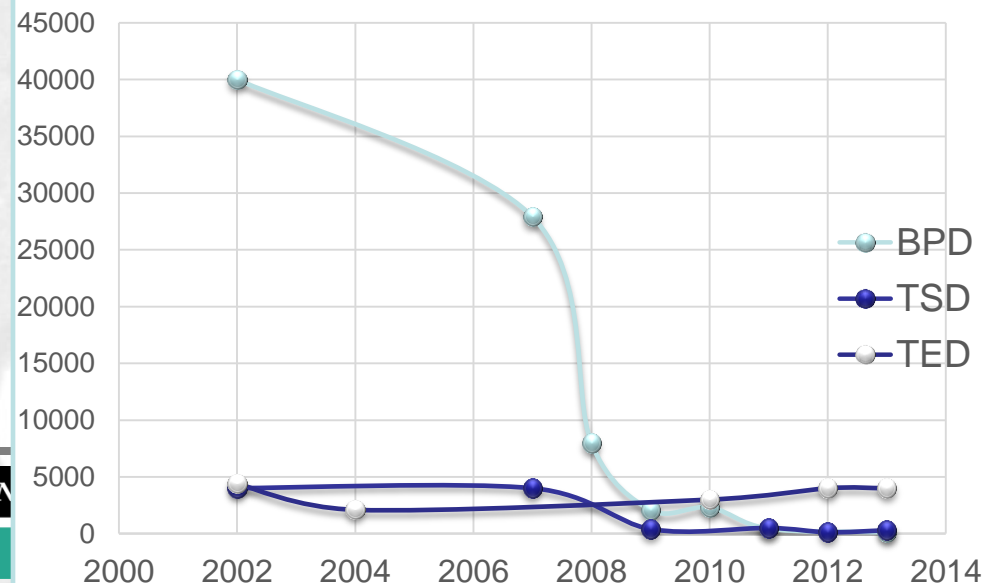
## TSD Density(cm-2)



**BPD and TSD density has been significantly reduced in the past 12 years or so. TED density has remained quite flat**

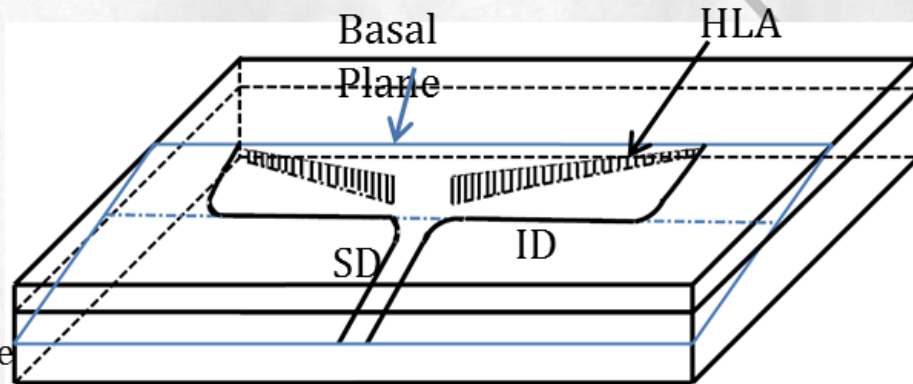
1c TSDs

## Dislocation Density(cm-2)





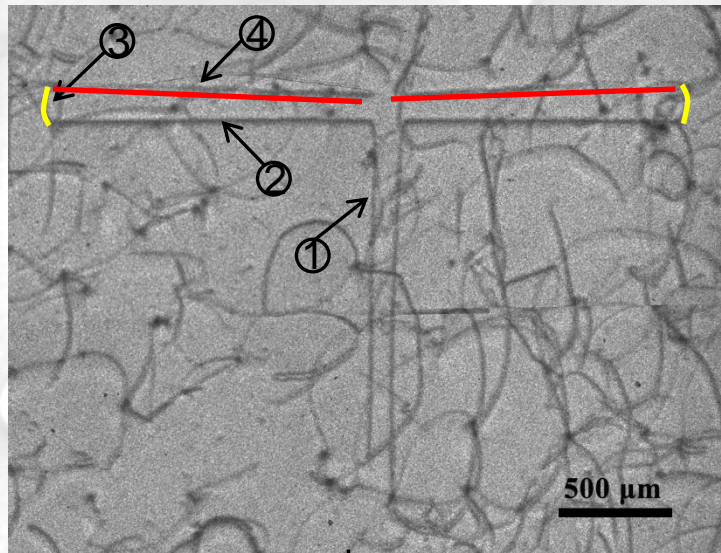
# Stress Relaxation During Homo-Epitaxy Interfacial Dislocations (ID) & Half-loop Arrays (HLAs)



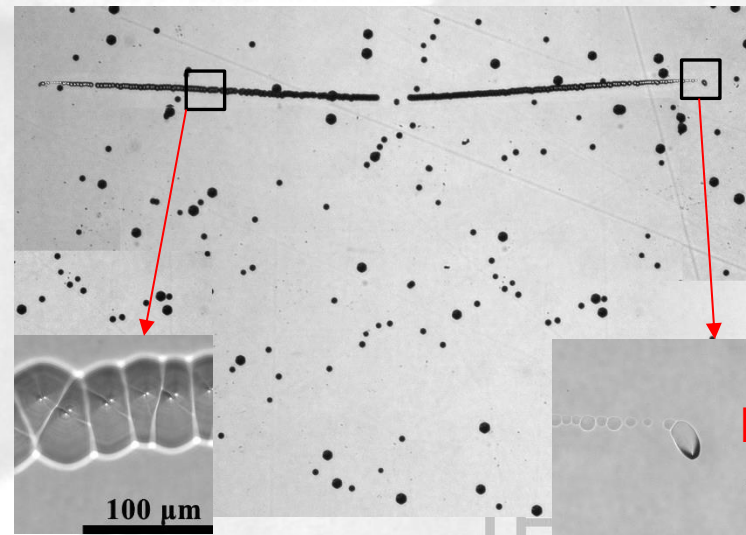
• **The Defect Configuration in question is composed of four parts:**

- (1) Screw BPD with the Burgers vector of  $1/3[11-20]$  (parallel to off-cut direction) in the substrate.
- (2) Straight edge-type BPD segments along the epilayer/substrate interface.
- (3) BPD segment side-gliding in the epilayer.
- (4) Half-loop arrays which have the same Burgers vector as interfacial dislocations.

*H. Wang, F. Wu, M. Dudley, et al. 2013 ICSCRM presentation, Miyazaki.*



$g=11-20$



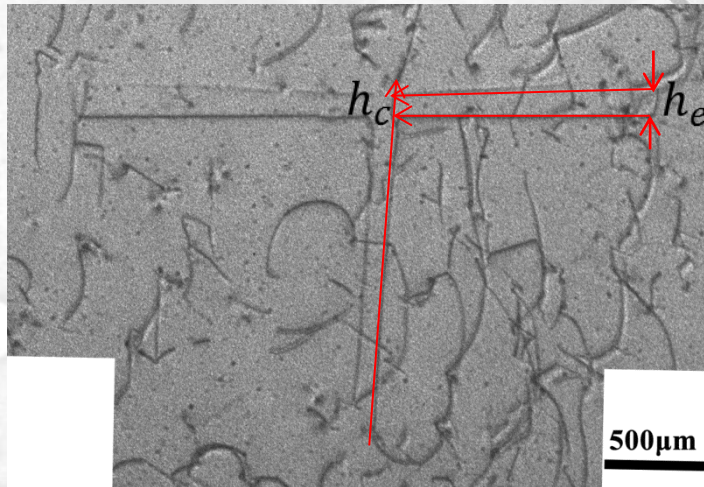
Downstep

Etch pits



# Measurement of Critical Thickness using X-ray Topography

## white beam transmission topography



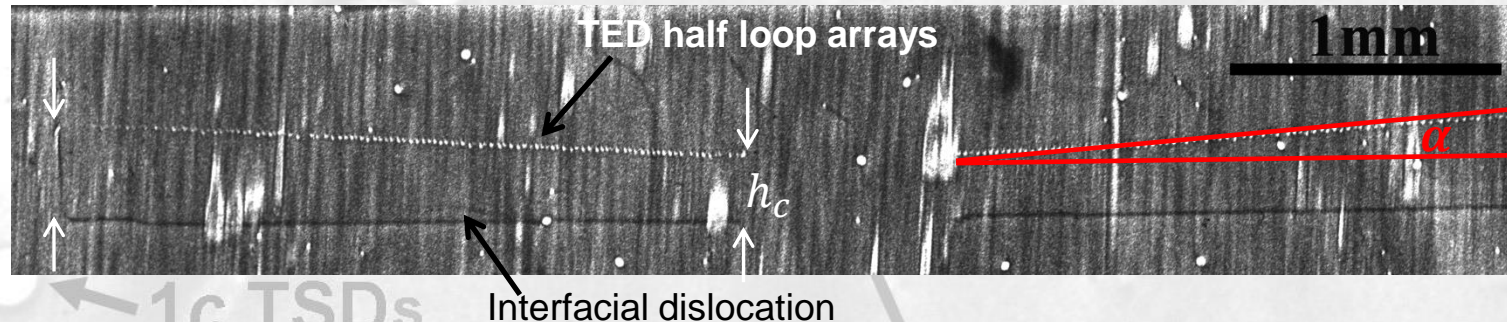
$v_0$ : epilayer growth rate;  
 $v_g$ : interfacial dislocation gliding rate.

$$\frac{v_g}{v_0} = \tan \alpha$$

Thus if we know the growth velocity, the gliding velocity of the dislocation could be calculated.

H. Wang, F. Wu., M. Dudley, B. Raghothamachar, G. Chung, J. Zhang, B. Thomas, E.K. Sanchez, S.G. Mueller, D. Hansen, and M. J. Loboda, "in *Silicon Carbide and Related Materials 2013*, H. Okumura, H. Harima, T. Kimoto, M. Yoshimoto, H. Watanabe, T. Hatayama, H. Matsuura, T. Funaki and Y. Sano (Eds.), **Materials Science Forum**, **778-780**, 328-331, (2014).

## Monochromatic grazing topography on etched wafer



↑ Downstep

- $h_e$  is corresponding to the thickness of the epi (12.5  $\mu\text{m}$ ) projected on the wafer surface,  $h_c$  is corresponding to the projection of critical thickness. And  $h_c$  is estimated around **8  $\mu\text{m}$** . If the thickness of the epilayer is smaller than 8  $\mu\text{m}$ , then the formation of IDs and HAPs could be avoided.

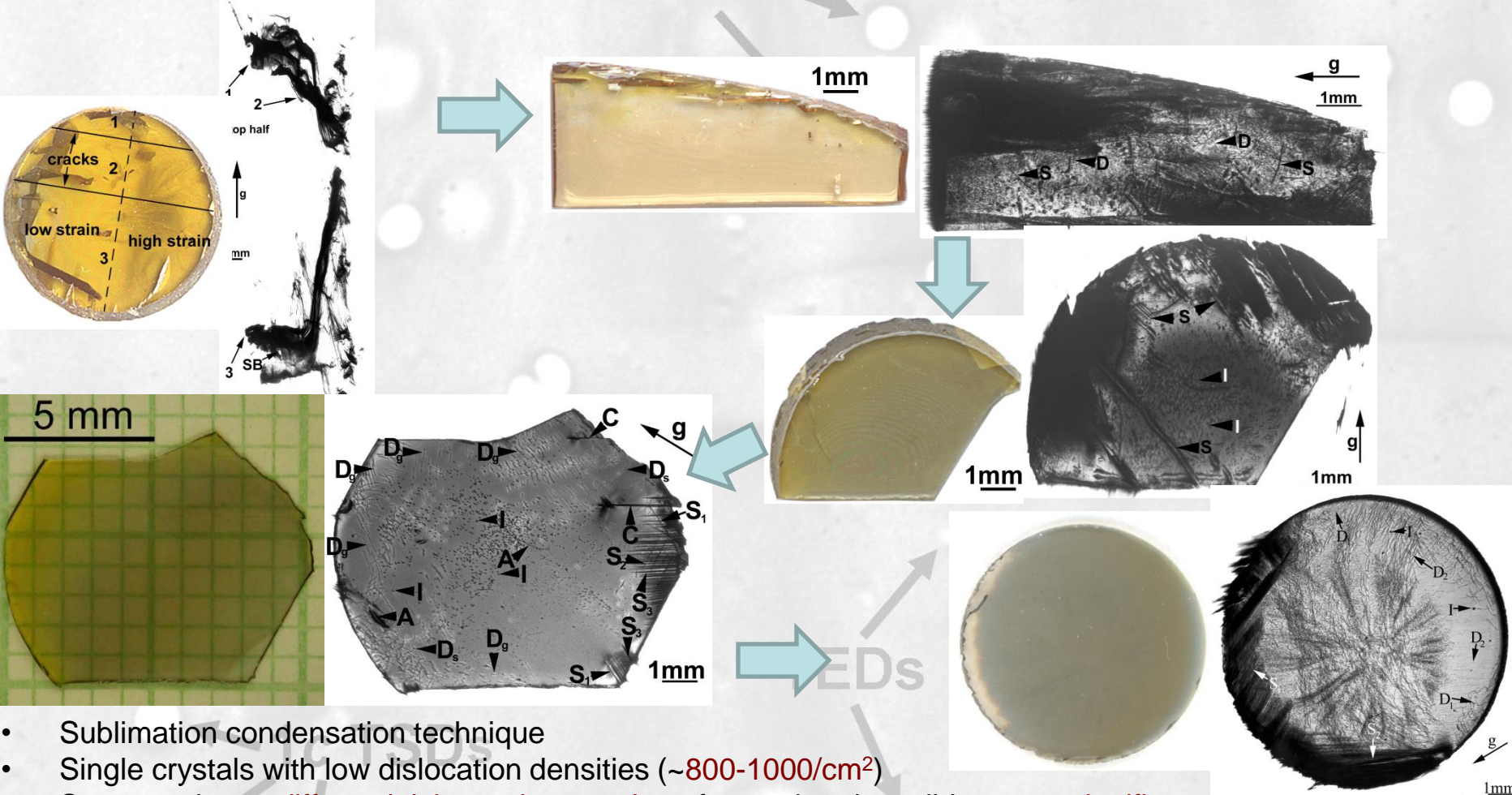
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# Bulk III-Nitride Electronics

- Wide band gap III-nitride semiconductor devices (GaN, AlN, InN)
  - Electronic devices capable of operation at elevated temperatures, high power or high frequency
  - Short wavelength optoelectronic devices (solar blind UV light detectors, UV laser diodes & LEDs)
  - Solid state lighting applications
- Devices on Bulk substrates
  - better performance and reliability of devices
  - simplify fabrication process and lower cost
- Aluminum nitride crystal growth
  - sublimation process
- Gallium nitride crystal growth
  - ammonothermal growth
  - sublimation process
- Requirements: Large area, low defect density single crystal substrates
  - Study as-grown boules and substrates to map defect types and their distribution to gain insight into defect nucleation mechanisms and propagation of defects
  - Develop growth strategies to minimize or eliminate defects

m nitride

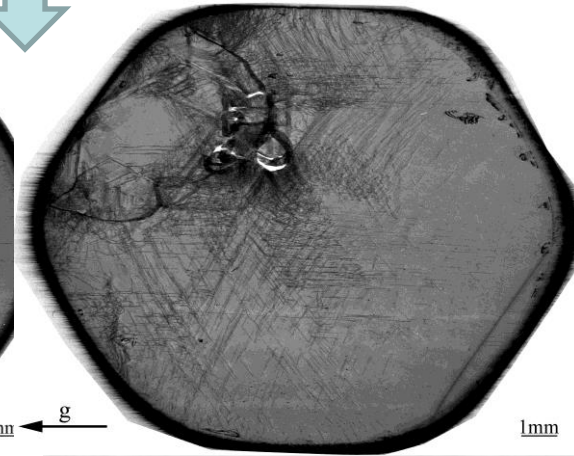
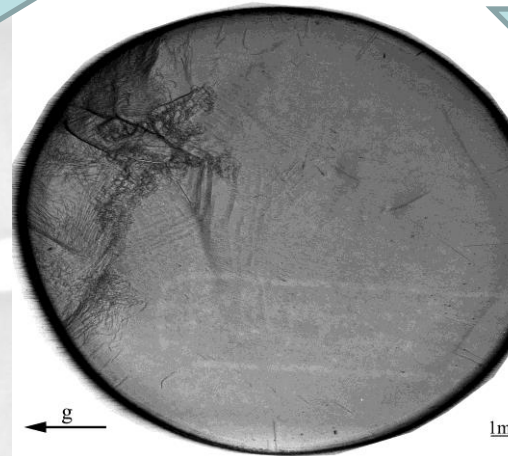
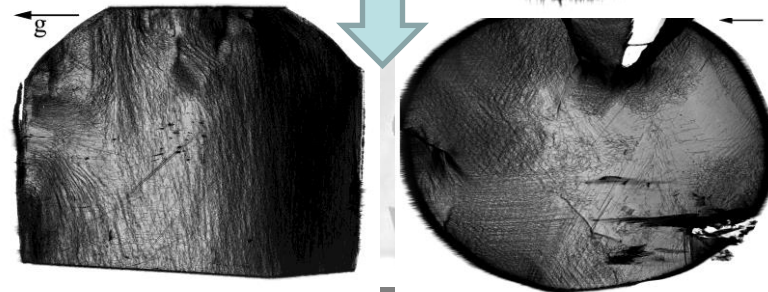
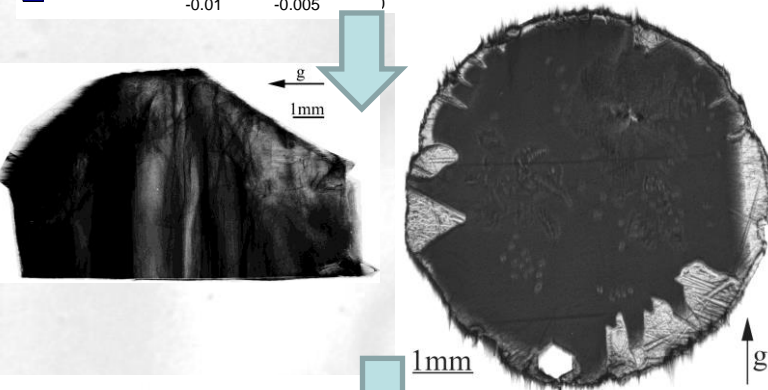
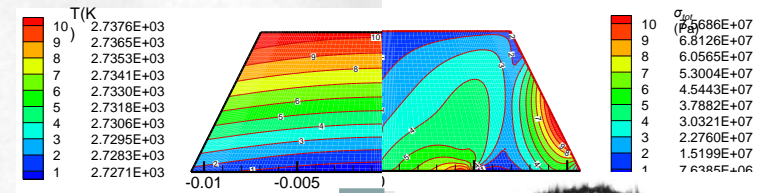
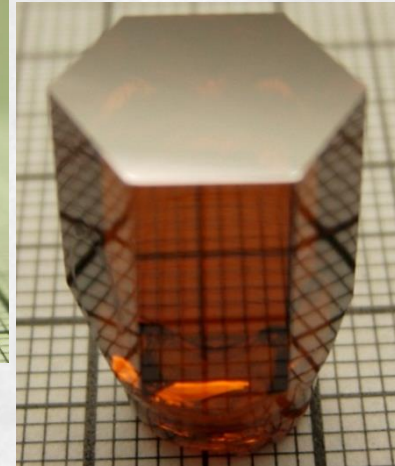
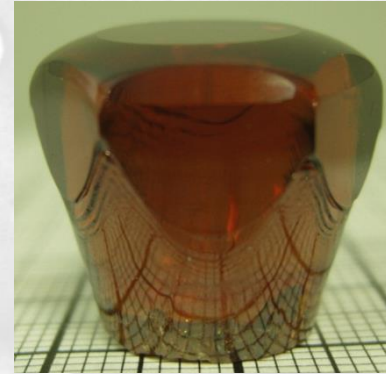
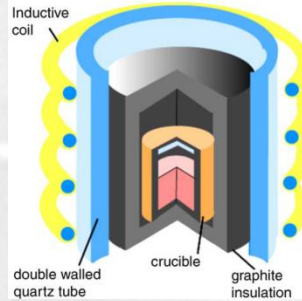
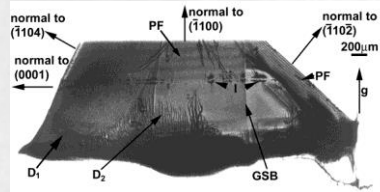
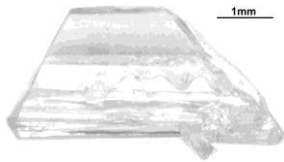


- Sublimation condensation technique
- Single crystals with low dislocation densities ( $\sim 800\text{-}1000/\text{cm}^2$ )
- Stresses due to **differential thermal expansion** of crystal and crucible cause **significant deformation** with basal plane slip at higher post-growth temperatures and prismatic slip and cracking at lower temperature



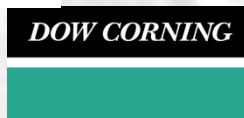
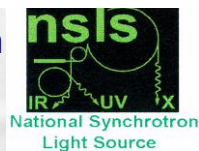
# Aluminum nitride electronics

1c



TEDs

**Stony Brook Synchrotron  
Topography Facility  
Beamline X-19C**



We help you  
invent the future.™



**Department of Materials  
Science & Engineering**

# Sapphire for Solid State Lighting Applications

- Advantages of solid state lighting
  - High energy efficacy
- High system efficiency (directional light):
  - 70 – 85 % luminaire efficiency
- Long lifetime:
  - 30,000 – 50,000 hrs of life
- Color and color tunability
- Easy switching and dimming
- Instant on/off
- Mercury-free
- High functionality
- Freedom of design: compact light sources, high brightness

(Source: OSRAM)

**C-plane oriented sapphire** is substrate of choice for the III-nitride based solid state lighting industry

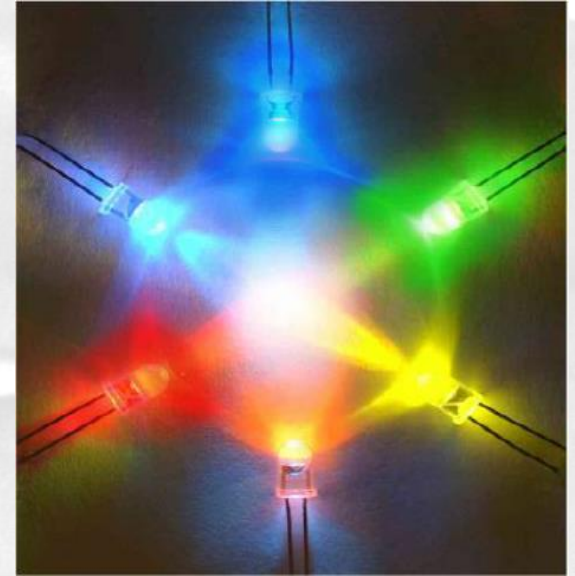


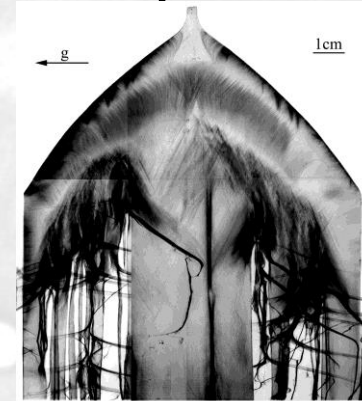
Photo: E.F. Schubert  
Rensselaer Polytechnic



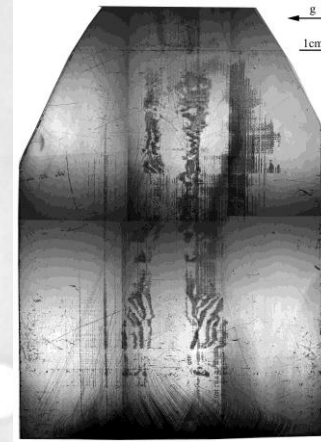
# Sapphire for Solid State Lighting Applications

## EFG growth technique

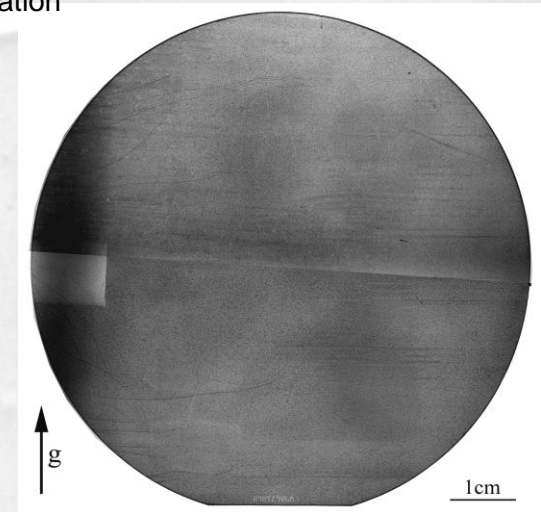
- Edge-defined film-fed growth (EFG) technique
  - Wafers can be directly obtained with minimal processing
  - Scalable to large widths (upto 18")
  - Simultaneous growth of multiple ribbons
- To develop the EFG technique for C-plane growth, a series of growth runs with concomitant structural characterization by SWBXT was carried out. Analysis of defect distribution and appropriate feedback to modify growth conditions to eliminate defects enabled the growth of nearly-defect free ribbons.



EFG-grown ribbon  
before growth  
optimization



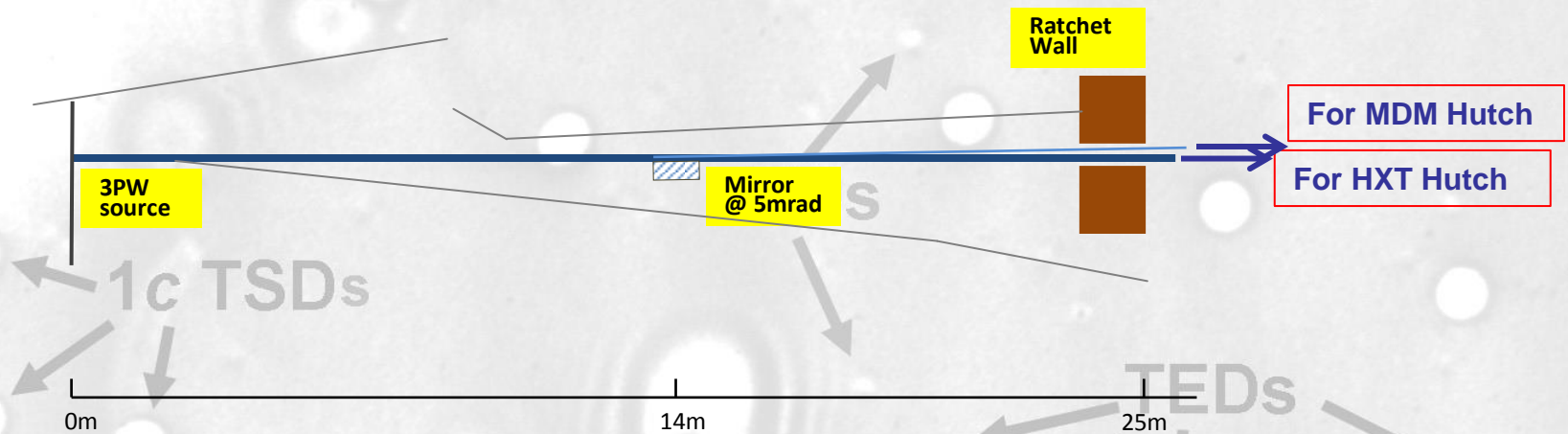
EFG-grown ribbon after  
growth optimization



Dislocation-free sapphire substrate  
obtained by EFG technique

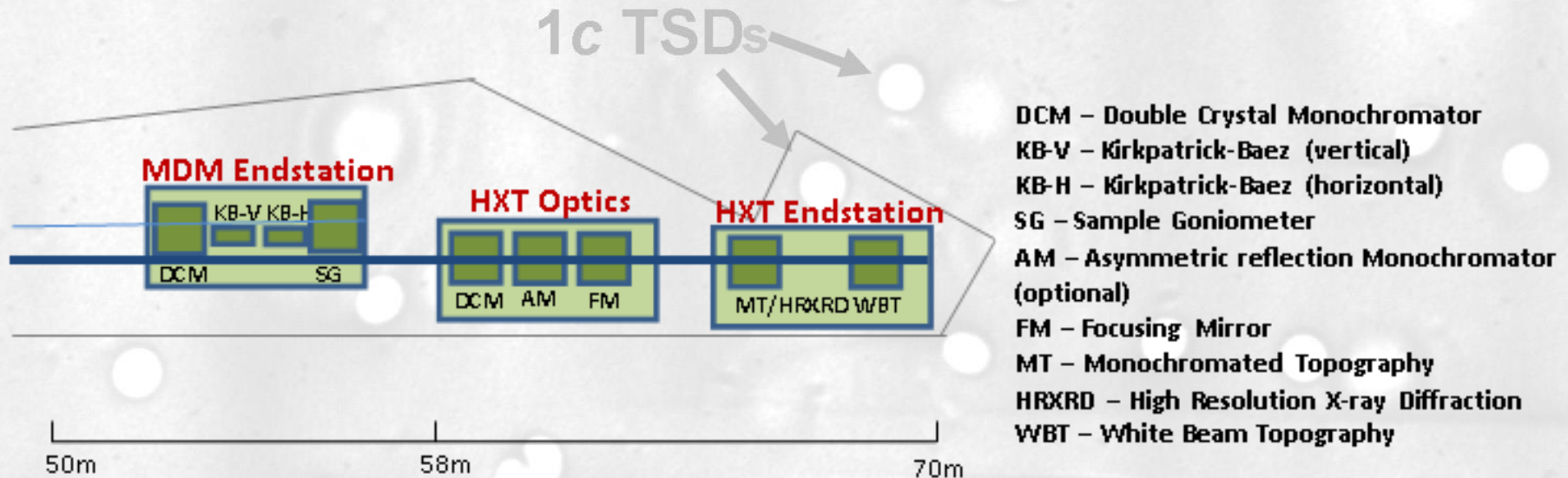
# HXT and MDM beamline layouts @ NSLS-II

- Beam from 3PW source will be shared by HXT and MDM hutches at NSLS-II
- Monochromatic/White Beam X-ray Topography and High Resolution Diffraction Beamline (HXT)
  - Prof. Michael Dudley (spokesperson), Stony Brook University
- MDM (Micronscale Detector Mapping (MDM) for Investigation of the Non-uniformity in the Gamma-ray Response of Large Area/Volume Radiation
  - Dr. Ralph James (spokesperson), Nonproliferation and National Security Department, BNL
- A Mirror will be used for Deflecting Portion of Beam for MDM Beamline (**0.18mrad of 3mrad** will be deflected)



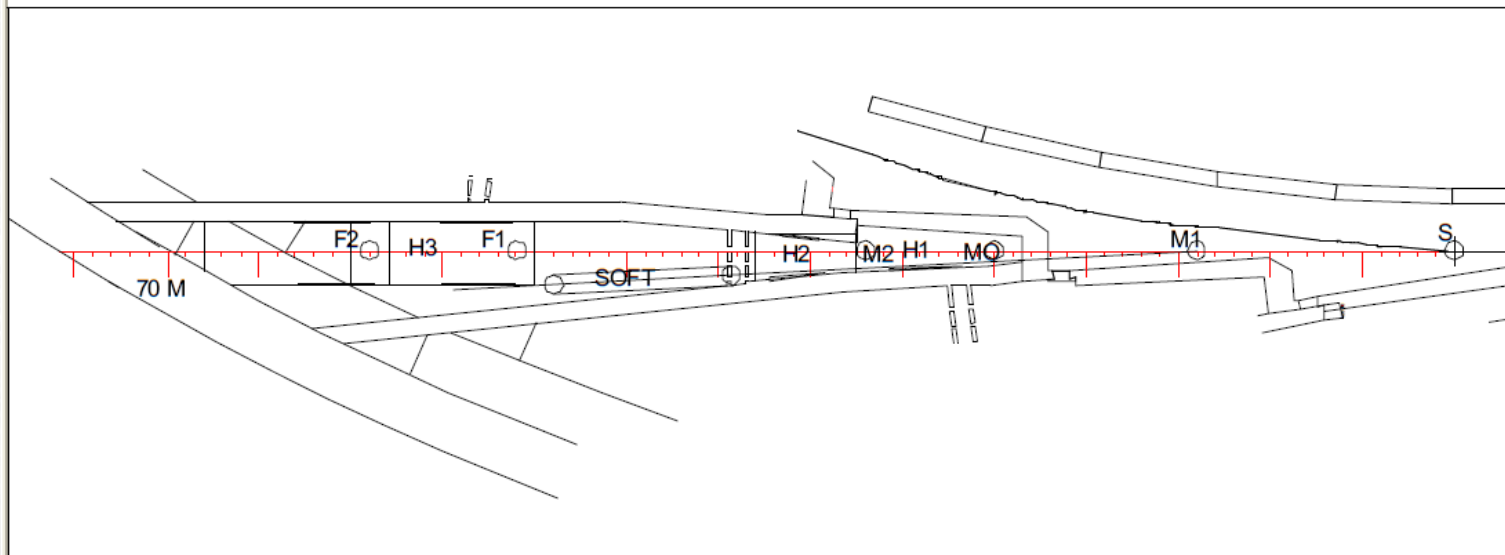


# HXT and MDM beamline layouts @ NSLS-II



- Water cooled channel-cut **Si(111) monochromator** (with weak link)
- **Asymmetric monochromator** for spreading the vertical beamsizes to improve coverage of large samples as well as to allow for slight detuning of the crystals to remove the unwanted effects of harmonics
- **Focusing mirror** for triple axis imaging, small beamsizes spatial mapping of rocking curves, grazing-incidence diffraction and reflectivity measurements
- Accurate **calibrated slits** (down to a few microns in width) to allow for section topography imaging as well as to facilitate small sizes for high resolution diffraction measurements
- **White beam slits** will also optionally be available in the optics hutch to control beam size.

# MID Beamline Layout @ NSLS-II



Proposed MID beamline layout.

Ruler is provided with tick marks for each meter from the source.

Key elements are indicated by the following symbols:

S. 3PW source

M1. Horizontal collection mirror for soft x-ray side branch

MO. DCM/DMM monochromator for main branch

H1,2,3. Test hutches 1-3, each capable of white or monochromatic beam component testing

M2. Vertical (toroid) focusing mirror for main branch (removable)

F1. Position of M2 focus for 60 m beamline

F2. Position of M2 focus for 70 m beamline (preferred)

SOFT. Soft x-ray sidebranch with grating monochromator and vacuum test chamber

Appendix 2. Beamline Layout (large format)

- From Metrology and Instrumentation Development (MID) Beamline Development Proposal (2010), Jeffrey Keister (spokesperson)



# Summary

1c TSDs

## ● Our Working Model for Industry/University Collaborative Research at a DOE Synchrotron Light Source:

- Long Term Contracts.
- Short Term Projects
- Students Graduated.
- Papers Published

## ● Case Studies:

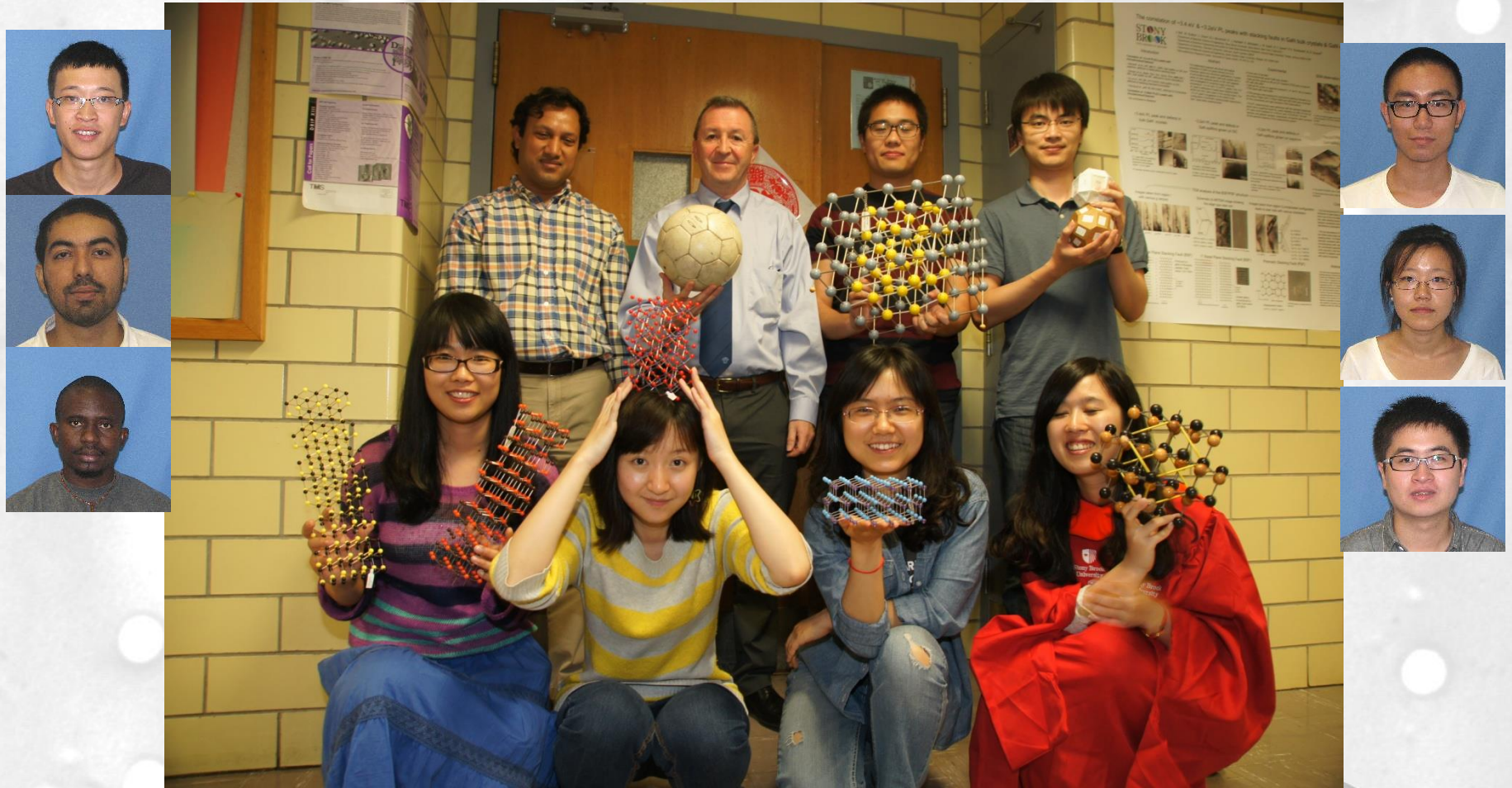
- Defect Density Reduction in SiC; Relaxation during Homo-Epitaxy :  
Funded by Cree; II-VI Inc.; Dow Corning
- Defect Analysis in Sapphire: Funded by St. Gobain; ARC Energy
- Defect Analysis in PVT Grown AlN Crystals: funded by Hexatech Inc.

## ● NSLS-II HXT Beamline Development Proposal Approved 2013

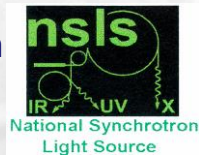
- Possible collaboration with MID beamline (NxtGen) along with MDM.

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# Thank You!



**Stony Brook Synchrotron  
Topography Facility  
Beamline X-19C**



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Science & Engineering**